Datasheet for the decision of 7 August 2008

Case Number: T 1497/05 - 3.3.10
Application Number: 99306704.0
Publication Number: 0985653
IPC: C07C 51/12
Language of the proceedings: EN
Title of invention: Carbonylation process
Patentee: BP Chemicals Limited
Opponent: Celanese Ltd.
Headword: Carbonylation process/BP
Relevant legal provisions: EPC Art. 56
Keyword: "Inventive step - yes - improvement - non-obvious solution"
Decisions cited: -
Catchword: -
DECISION

of the Technical Board of Appeal 3.3.10
of 7 August 2008

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Composition of the Board:
Chairman: R. Freimuth
Members: J. Mercey
F. Blumer
Summary of Facts and Submissions

I. The Appellant (Opponent) lodged an appeal on 7 December 2005 against the decision of the Opposition Division of 11 October 2005 rejecting the opposition against European patent No. 985 653 which was granted on the basis of ten claims, claim 1 of which reading as follows:

"A process for removing higher organic iodides, including hexyl iodide, from an acetic acid product obtained by carbonylating methanol and/or a reactive derivative thereof in the presence of a finite concentration of water, Group VIII noble metal catalyst, methyl iodide as co-catalyst, and optionally a catalyst promoter, which process includes the step of subjecting an aqueous composition comprising acetic acid and at least one higher organic iodide to distillation in a column, or section of a column, separating water overhead from a dry acetic acid fraction, wherein the water concentration on the feed tray of the column, or section of the column, is greater than 8% by weight and the water concentration in the head of the column, or section of the column, is greater than 70% by weight."

II. Notice of Opposition had been filed by the Appellant requesting revocation of the patent as granted in its entirety on the grounds of insufficient disclosure and lack of inventive step. Inter alia the following document was cited by the Opposition Division in the opposition proceedings:

III. The Opposition Division held that the invention was sufficiently disclosed and involved an inventive step over inter alia document (6), said document not giving any hint that a specific water concentration in the head of the column would result in reduced hexyl iodide concentration in the acetic acid produced.

IV. The Appellant submitted that the subject-matter of the present invention was not inventive over document (6), said document being concerned with a process for producing high purity acetic acid by removal of impurities such as hexyl iodide. More particularly, document (6) disclosed a process for producing acetic acid by carbonylating methanol, which process included the step of subjecting an aqueous composition comprising crude acetic acid from said reaction to distillation in a column, wherein water was separated from the top and an acetic acid fraction from the bottom or the side of the column. In Example 1 acetic acid containing 2.2 wt.% water and 4 ppm hexyl iodide was thus obtained. Document (6) taught that impurities in the crude acetic acid, such as hexyl iodide, formed an azeotrope with water. It further taught that impurities were driven to the top of the distillation column by water refluxed into the top of the distillation column when the concentration of water in the crude acetic acid was high, and that at reduced water concentrations the separability in the decanter at the top of the distillation column deteriorated such that this ability to drive impurities to the top of the column was impaired, the impurities as a result mixing with the acetic acid instead. Document (6) taught that water could be added not only to the first, light ends, distillation column described therein, but also to the
second, drying, column. Since document (6) taught the advantageous use of adding water to a distillation column in order to facilitate the removal of impurities, such as hexyl iodide, the determination of the particular concentration of water required in the head of the column in order to achieve optimal removal was merely a matter of routine experimentation for the skilled person.

V. The Respondent (Patent Proprietor) submitted that starting from document (6), the problem to be solved by the patent in suit was to provide a process for preparing dry acetic acid which had reduced hexyl iodide content. Controlling the water profile concentration of the distillation column such that the water concentration in the heads was greater than 70 wt.% resulted in a significant decrease in the level of hexyl iodide in the acetic acid (5 ppb instead of 20 to 120 ppb), as demonstrated by the Comparison Test and Example in the specification of the patent in suit. Given that document (6) neither explicitly taught that hexyl iodide formed an azeotrope with water, let alone in the system of the claimed process, and was completely silent with regard to the concentration of water in the head of the distillation column, the skilled person would not have arrived at the claimed process without exercising inventive ingenuity.

VI. The Appellant requested that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the appeal be dismissed.
VII. At the end of the oral proceedings held on 7 August 2008, the decision of the Board was announced.

**Reasons for the Decision**

1. The appeal is admissible.

2. **Inventive step**

2.1 According to the established jurisprudence of the Boards of Appeal it is necessary, in order to assess inventive step, to establish the closest state of the art, to determine in the light thereof the technical problem which the invention addresses and successfully solves, and to examine the obviousness of the claimed solution to this problem in view of the state of the art. This "problem-solution approach" ensures assessing inventive step on an objective basis and avoids an ex post facto analysis.

2.2 The patent in suit is directed to a process for removing higher organic iodides from aqueous acetic acid. A similar process already belongs to the state of the art in that document (6) (see claim 1) describes a process for producing high purity acetic acid by reacting methanol with carbon monoxide in the presence of a Group VIII metal catalyst, methyl iodide and water, which process includes the step of subjecting an aqueous composition comprising crude acetic acid from said reaction to distillation in a column, wherein water is separated from the top and an acetic acid fraction from the bottom or the side of the column. In
Example 1 acetic acid containing 2.2 wt.% water and 4 ppm hexyl iodide is thus obtained.

Thus, the Board considers, in agreement with the Appellant and the Respondent, that the process of document (6) represents the closest state of the art and, hence, takes it as the starting point when assessing inventive step.

2.3 In view of this state of the art, the problem underlying the patent in suit, as formulated by the Appellant at the oral proceedings and indicated on column 3, lines 34 to 40 of the specification of the patent in suit, consists in providing an improved process for preparing dry acetic acid with a reduced concentration of higher iodides.

2.4 As the solution to this problem, the patent in suit proposes a distillation process as defined in claim 1 comprising the water concentration on the feed tray of the column, or section of the column, being greater than 8% by weight and the water concentration in the head of the column, and/or section of the column, being greater than 70% by weight.

2.5 The Example and Comparison Test of the specification of the patent in suit demonstrate that the claimed process results in an acetic acid product with a lower hexyl iodide concentration (namely 5 ppb instead of 20 to 120 ppb). However, these examples differ only by virtue of the water concentration in the head of the column being either greater or less than 70% by weight, respectively, the water concentration on the feed tray of the column being in each case the same.
As such, a causal link between the reduction in the concentration of hexyl iodide has been established only for the feature of the water concentration in the head of the column being greater than 70% by weight, such that only this feature characterises the invention.

The Appellant argued that it had not been convincingly shown that the problem had been successfully solved, since the water concentrations in the head of the columns of the Example and Comparison Test were given as broad, average ranges (35 to 68 wt.% and 70 to 85 wt.% respectively), it not being apparent for how long the column was operated at any particular water concentration, such that the results were meaningless and the comparison not pertinent. However, the Board considers that for an industrial process operated over several months, fluctuations are not unusual. In any case, the lowest concentration and hence best result, namely 20 ppb of hexyl iodide, achieved in the Comparison Test is still four times greater than the average value achieved in the example according to the invention, such that it is plausible that the limit of 70% by weight for the water concentration in the head of the column is critical and the comparison is fair. The Board is thus satisfied that the problem underlying the patent in suit has been successfully solved.

2.6 Finally, it remains to be decided whether or not the proposed solution to the problem underlying the disputed patent is obvious in view of the cited prior art.
2.6.1 Neither party cited any other prior art document during the appeal proceedings, such that the only question which needs to be answered is whether or not document (6) teaches the skilled person to use a high concentration of water in the head of a distillation column, in particular greater than 70% by weight, in order to reduce the hexyl iodide concentration in an acetic acid fraction withdrawn therefrom.

2.6.2 The Board holds that document (6) does not contain such a teaching. The Appellant, however, although acknowledging that said document does not contain such an explicit teaching, argued that it did nevertheless direct the skilled person to use a high concentration of water in the head of a distillation column in order to solve the problem underlying the patent in suit.

2.6.3 In support of its argument, the Appellant first submitted that document (6) (cf. col. 3, lines 5 to 12 and 20 to 22) taught that impurities present in the crude acetic acid, including hexyl iodide, formed an azeotrope with water. The Board concurs with the Appellant in this respect, since, even if certain impurities taught by document (6) as forming an azeotrope with water, for example, acetaldehyde, did not in fact do so, as contended by the Respondent, the skilled person would nevertheless have followed the clear teaching of document (6) with a reasonable expectation of success i.e. of forming an azeotrope. The Board cannot concur with the Respondent's argument that due to some purported uncertainty about the predictability of success, the skilled person would not have followed said teaching, since nothing has been submitted by the Respondent from which the Board could
reasonably conclude that the skilled person would have been deterred from following the teaching of the art. It was only necessary for him to confirm experimentally by routine work that hexyl iodide does indeed form an azeotrope with water.

2.6.4 The Appellant further submitted that document (6) (cf. col. 6, lines 21 to 25) taught that when the water concentration in the carbonylation reaction liquid was lowered, the separability in the decanter at the top of the distillation column deteriorated, thereby leading the skilled person to increase the water concentration. However, this teaching concerns a mechanical separation in a decanter and is irrelevant to a thermal separation in a distillation column as claimed by the patent in suit. The Appellant also submitted that document (6) (cf. col. 6, lines 25 to 33) taught that impurities were driven to the top of the distillation column by water refluxed into the top of the distillation column when the concentration of water in the crude acetic acid from the carbonylation reaction was high. Since hexyl iodide formed an azeotrope with water, the skilled person would be prompted by this teaching to use an appropriately high concentration of water in the distillation column in order to remove higher iodides. The Appellant also referred in this respect to the Respondent's submission of 7 October 2004 before the Opposition Division, wherein it was stated that the skilled man would know that the water concentration in a column was achieved by controlling the amount of reflux thereto. However, this teaching in document (6), although indeed referring to water concentration, concerns the concentration of water in the output from the carbonylation reaction, said output being the feed...
to the distillation column, the water concentration in the column feed, however, not corresponding to the water concentration in the head of the distillation column. And although this teaching in document (6), as well as the submission of the Respondent before the Opposition Division, also refers to water refluxed into the top of the distillation column, it does not address the concentration of water in the head of the column and in particular does not hint towards a threshold value of 70% by weight, let alone any impact the water concentration in the head of the column may have on the extent of removal of each individual impurity, let alone of hexyl iodide.

2.6.5 Finally, the Appellant submitted that document (6) (cf. col. 7, lines 14 to 24) taught the addition of water to the feed of both a first, light ends, distillation column as well as to a second, drying, column, such that the advantage of adding water, as taught at col. 6, lines 21 to 33 (cf. point 2.6.4 supra), was equally applicable to a distillation in a drying column, the process of the patent in suit being carried out in a drying column. However, as indicated in point 2.6.4 supra, the water concentration in the column feed, however, does not correspond to the water concentration in the head of the distillation column, such that a teaching concerning the addition of water to the column feed cannot render the proposed solution, namely the water concentration in the head of the column being greater than 70% by weight, obvious.

2.7 To summarise, in the Board's judgement, document (6) does not render the claimed invention obvious.
2.8 For these reasons, the Board concludes that the subject-matter of claim 1, and by the same token that of dependent claims 2 to 10, involves an inventive step within the meaning of Articles 52(1) and 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

P. Cremona R. Freimuth